J. Synchrotron Rad. (1998). **5**, 1111–1113

Submicrometre-area high-energy-resolution photoelectron spectroscopy system

Takanori Kiyokura,[a]* Fumihiko Maeda,[a] Yoshio Watanabe,[a] Eiji Shigemasa,[b] Akira Yagishita,[b] Masaharu Oshima,[c] Yoshinori Iketaki[d] and Yoshiaki Horikawa[d]

[a]NTT Basic Research Laboratories, 3-1 Morinosato Wakamiya, Atsugi-shi, Kanagawa 243-01, Japan, [b]High Energy Accelerator Research Organization, 1-1 Oho, Tsukuba-shi, Ibaraki 305, Japan, [c]The University of Tokyo, 7-3-1 Hongo, Bunkyo-ku, Tokyo 113, Japan, and [d]Olympus Optical Company Ltd, 2-3 Kuboyama-cho, Hachioji-shi, Tokyo 192, Japan. E-mail: kiyokura@will.brl.ntt.co.jp

(*Received 4 August 1997; accepted 21 October 1997*)

A submicrometre-area photoelectron spectroscopy system that uses a multi-layer-coated Schwarzschild objective as the soft X-ray microbeam optics has been developed. The system is located at an undulator beamline (BL-16U) at the Photon Factory in the High Energy Accelerator Research Organization. By knife-edge measurement, the microbeam size was estimated to be 160 nm at the sample position using a 25–75% criterion. Photoelectron spectral measurements revealed that the Fermi edge width was 0.12 eV, which means that the instrumental resolution was 0.05 eV, after removing the natural broadening of the Fermi edge at room temperature. This system offers both high energy resolution and high spatial resolution.

Keywords: photoelectron spectroscopy; microbeams; Schwarzschild objectives; soft X-rays; undulators.

1. Introduction

When a microbeam is formed as the focused image of a pinhole by microbeam optics, a smaller-diameter pinhole is required to obtain a smaller microbeam. Although the focused beam size becomes smaller when we use a smaller diameter pinhole, the photon flux decreases in proportion to the square of the pinhole diameter. Therefore, to achieve both small beam size and high photon flux at the same time, a large-diameter pinhole and microbeam optics with a high demagnification ratio are required. We have developed a photoelectron spectroscopy (PES) system with submicrometre spatial resolution, using a 10 μm-diameter pinhole and a Schwarzschild objective (SO) with a 1/224 demagnification ratio, which is much larger than the other microbeam optics used in microbeam PES systems (Ade, 1992; Casalis *et al.*, 1995; Johansson *et al.*, 1995; Ng *et al.*, 1994; Voss *et al.*, 1992). Furthermore, by using this SO with a high-energy-resolution monochromator, our system was able to attain both high energy resolution and small beam size simultaneously. In this paper we describe the performance of our submicrometre-area photoelectron spectroscopy system, and we discuss the results of evaluating microbeam size by knife-edge measurement, and the results of evaluating instrumental energy resolution by Au Fermi-edge photoelectron spectral measurement.

2. Instrumentation

The submicrometre-area high-energy-resolution photoelectron spectroscopy system consists of two key devices, namely the undulator beamline and the submicrometre-area PES apparatus (Fig. 1). The undulator beamline consists of an undulator light source, a monochromator and refocusing mirrors. The submicrometre-area PES apparatus consists of a sample stage, an electron energy analyser and microbeam optics, which consist of a pinhole slit and an SO.

2.1. *Beamline*

An undulator with 26 periods and a period length of 120 mm provides high photon flux with low emittance in the soft X-ray region, making it suitable for microbeam formation. The basic design of the beamline, including ray-tracing calculations, was presented by Shigemasa *et al.* (1995). The beam from the undulator is monochromated by a 24 m spherical grating monochromator with moving exit slit, which is called a 'Dragon-type' monochromator (Chen, 1987). We used a grating of 900 lines mm^{-1} to disperse the undulator beam. The beam was then focused onto the pinhole slit by the refocusing mirrors, which are Kirkpatrick–Baez-mounted bent cylindrical mirrors. Ray-tracing simulation showed that the full width at half-maximum of the beam size on the pinhole slit was 0.03 mm (V) × 0.2 mm (H).

2.2. *Submicrometre-area PES apparatus*

Fig. 2 shows a side view of the submicrometre-area PES apparatus. The system consists of a pinhole slit, a Schwarzschild objective, a sample stage, an optical bench, a multi-channel plate (MCP), a three-axis optical bench controller and a 200 mm-radius hemispherical electron energy analyser.

2.2.1. *Microbeam optics and alignment.* A stainless-steel multi-pinhole is mounted at the focal point, forming a soft X-ray source for the SO. The pinhole size can be changed by selecting one of various pinhole diameters: 300, 100, 30 or 10 μm. The pinhole slit

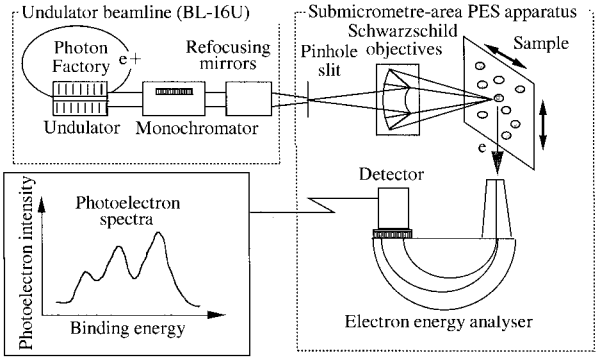

Figure 1
A schematic view of the submicrometre-area high-energy-resolution photoelectron spectroscopy system. BL-16U is the undulator source for the Photon Factory. The monochromator is a high-energy-resolution spherical grating monochromator, the so-called 'Dragon-type'. The Schwarzschild objective is a multi-layer-coated soft X-ray optics system with demagnification ratio of 1/224. The electron energy analyser is a high-energy-resolution hemispherical electron energy analyser with 200 mm analyser radius.

acts as a pseudo light source for the SO. The distance between the pinhole slit and the focal point of the SO, *i.e.* image–object (IO) distance, is 1000 mm. The distance between the frame of the SO and the sample position, *i.e.* working distance, is 10 mm. The obstruction ratio is 0.57, and the numerical aperture is 0.235. The SO consists of a normal-incidence convex and concave mirror. The multilayer on the convex and concave mirrors is coated with 41 layers of Mo/Si (3.2 nm/3.9 nm); therefore, the SO only passes photons near 89 eV. The demagnification ratio is 1/224 at an IO distance of 1000 mm. This high demagnification ratio increases the throughput of the optics, because a larger-diameter pinhole slit can be used. The specific properties of the Schwarzschild objective have been reported by Iketaki *et al.* (1996).

Since the beamline for this apparatus is used by time-sharing, we should align the optics every machine time. Therefore, ease of optical alignment should be given priority. To simplify optical alignment, the pinhole, SO and sample stage were optically aligned and set on the same optical bench. The relative position between the pinhole and SO was previously aligned. By using a manually controlled three-axis micrometer optical bench, we could align the rays from the beamline and the optical axis of the optical bench.

2.2.2. *Sample positioning and submicrometre-area photoelectron spectroscopy.* The top view of the experimental set-up for submicrometre-area photoelectron spectroscopy is shown in Fig. 3. Only soft X-rays of 89.2 eV (13.9 nm) can be used because of the multilayer reflectivity bandpass. The sample stage consists of three parts: a tube-type piezoelectric scanner for fine sample scanning, a two-axis bimorph motor sample stage for coarse sample positioning, and a one-axis piezotranslator with a strain gauge for fine sample positioning. Coarse positioning was performed by the two-axis bimorph motor sample stage, and fine positioning was performed by the one-axis piezotranslator with the strain gauge to attain a positioning accuracy of 40 nm. After finding the focal point, the tube-type piezoelectric scanner was driven and the sample surface was scanned 45° from the beam direction. If the sample is cleaved, the knife-edge method can be used to find the focal point of the SO. In this method, the cleaved sample edge is used as the knife-edge. Then the knife-edge curve, which is the dependence of the knife-edge position on the transmitted soft X-ray intensity, is measured. The transmitted soft X-ray intensity is measured as an anode current for a multi-channel plate. A knife-edge position with minimal curve width is

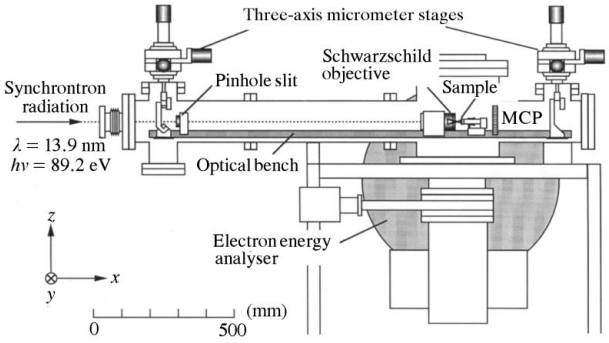

Figure 2
Side view of the submicrometre-area photoelectron spectroscopy apparatus. For ease of optical alignment, the pinhole slit, Schwarzschild objective and sample stage are optically aligned and set on the same optical bench. The direction of the optical bench can be controlled from outside the vacuum chamber, to align the optical axis and the synchrotron radiation beam.

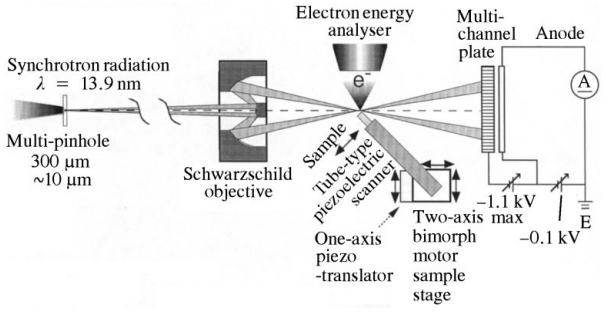

Figure 3
Top view of the sample stage and the measurement system for the apparatus. Coarse sample positioning is performed using the two-axis stage, and fine positioning is performed using the piezoelectric tube scanner.

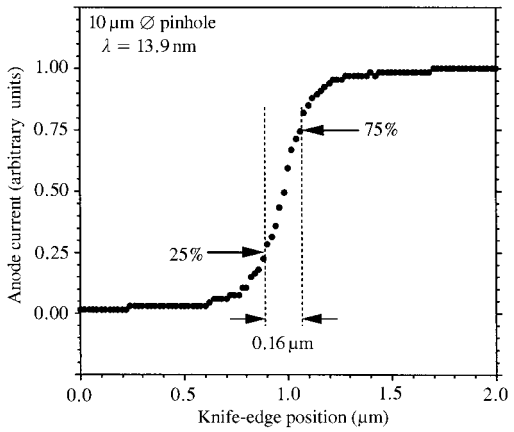

Figure 4
The knife-edge response curve of the focused beam. The knife edge is fabricated by KOH-etching of the Si wafer. Al is deposited on the edge to obstruct soft X-rays. The beam size is 0.16 μm using a 25–75% criterion.

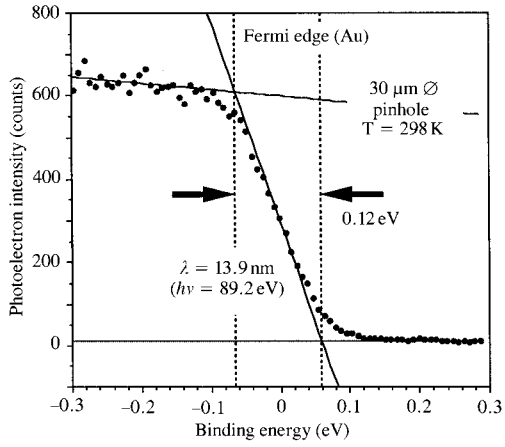

Figure 5
Total electron energy resolution measured by the photoelectron spectra of the Au Fermi edge. The combined energy resolution of the monochromator and the electron energy analyser was estimated to be about 0.05 eV by removing the natural broadening of the Fermi edge at room temperature.

considered to be the focal point. If the sample cannot be cleaved, we should search for the sample position where the photoelectron intensity curve of some small structure edge on the sample has the strongest contrast in order to find the focus point. Using these procedures, submicrometre-area photoelectron spectroscopy can be performed at any point that the tube-type piezoelectric scanner can reach.

3. Test experiments

To evaluate the spatial resolution of the system, we measured the beam size by placing a knife edge at the sample position. A 10 μm-diameter pinhole and a KOH-etched Si-wafer knife edge were used. The knife edge was moved across the focused soft X-ray beam using the one-axis piezotranslator, and the change in intensity of the transmitted soft X-ray beam (knife-edge response curve) was measured. Fig. 4 shows the knife-edge response curve of the beam. The size of the beam was estimated to be 0.16 μm using a 25–75% criterion. This value is 3.6 times the value of 0.045 μm which was simply calculated from the demagnification ratio of the SO. This result may have been influenced by vibrations in the apparatus, since the system does not have an isolator to reduce vibrations.

To determine the combined energy resolution for the monochromator of the beamline and the electron energy analyser, the Fermi edge photoelectron spectra of a clean Au plate were measured as shown in Fig. 5. The pinhole diameter was 30 μm, and the photon flux was estimated to be 3×10^9 photons s^{-1} from photocurrent measurement. The acquisition time for this spectrum was about 6 min. In Fig. 5 the Fermi edge width is 0.12 eV. Using this value, the combined energy resolution for the monochromator and the electron energy analyzer was estimated to be about 0.05 eV by removing the natural broadening of the Fermi edge at room temperature.

4. Summary

Our submicrometre-area photoelectron spectroscopy system with high energy resolution provided a highly monochromated soft X-ray beam with a diameter of 0.16 μm on the sample surface. The photoelectron spectra for the Fermi edge spectrum were measured using this soft X-ray beam. The Fermi edge width was 0.12 eV at room temperature, and instrumental resolution was estimated to be 0.05 eV. The high energy resolution and high throughput optics of the system made it well suited for the high-resolution spectroscopy and imaging of samples with spatial inhomogeneties in the submicrometre range. These values indicate the feasibility of using this spectroscopy system to study the electronic and chemical states of nanostructures, expected in future optical devices.

References

Ade, H. (1992). *Nucl. Instrum. Methods*, A**319**, 311–319.
Casalis, L., Jark, W., Kiskinova, M., Lonza, D., Melpignano, P., Morris, D., Rosei, R., Savoia, A., Abrami, A., Fava, C., Furlan, P., Pugliese, R., Vivoda, D., Sandrin, G., Wei, F.-Q., Contarini, S., DeAngelis, L., Gariazzo, C., Nataletti, P. & Morrison, G. R. (1995). *Rev. Sci. Instrum.* **66**, 4870–4875.
Chen, C. T. (1987). *Nucl. Instrum. Methods*, A**256**, 595–604.
Iketaki, Y., Horikawa, Y., Mochimaru, S., Nagai, K., Kiyokura, T., Oshima, M. & Yagishita, A. (1996). *J. Electron Spectrosc. Relat. Phenom.* **80**, 353–356.
Johansson, U., Nyholm, R., Tornevik, C. & Flodstrom, A. (1995). *Rev. Sci. Instrum.* **66**, 1398–1400.
Ng, W., Ray-Chaudhuri, A. K., Liang, S., Singh, S., Solak, H., Welnak, J., Cerrina, F., Margaritondo, G., Underwood, J. H., Kortright, J. B. & Perera, R. C. C. (1994). *Nucl. Instrum. Methods*, A**347**, 422–430.
Shigemasa, E., Yan, Y. & Yagishita, A. (1995). KEK Report 95–2. KEK, Tsukuba-shi, Ibaraki, 305, Japan.
Voss, J., Kunz, C., Moewes, A. & Storjohann, I. (1992). *Rev. Sci. Instrum.* **63**, 569–573.